

Bis(*O,O'*-di-*p*-tolylidithiophosphato- $\kappa^2$ S,S')nickel(II)

Shu-Shang Zhang,<sup>a</sup> Bo Yang,<sup>a</sup>  
Yan-Fang Wang,<sup>a</sup> Xue-Mei Li,<sup>a</sup>  
Kui Jiao,<sup>a</sup> Mohammad. B.  
Kassim<sup>b</sup> and Bohari. M. Yamin<sup>b\*</sup>

<sup>a</sup>College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao, Shandong 266042, People's Republic of China, and <sup>b</sup>School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi Selangor, Malaysia

Correspondence e-mail:  
bohari@pkkrisc.cc.ukm.my

## Key indicators

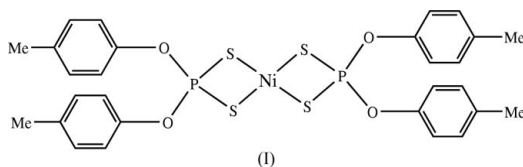
Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma$ (C–C) = 0.003 Å  
*R* factor = 0.030  
*wR* factor = 0.083  
Data-to-parameter ratio = 16.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, [Ni(C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>PS<sub>2</sub>)<sub>2</sub>], consists of discrete molecules with the Ni atom chelated by both S atoms of the *p*-tolylidithiophosphate ligands in a bidentate manner in a four-coordinate environment. The molecule has inversion symmetry about the Ni atom, and the geometry is nearly perfect square planar, with S–Ni–S chelate bond angles of 89.12 (2)°.

## Comment

Studies on metal–dithiophosphate and dithiophosphate complexes are nowadays driven by their biological properties such as fungicidal and antibacterial activity (Livingstone & Mihkelson, 1970) and in materials applications, such as additives in lubricating oils (Jones & Symes, 1971; Shiomi *et al.*, 1989). In the title compound, (I), the molecule is discrete and has a centre of inversion at the central Ni atom. The geometry at nickel is nearly a perfect square plane, with an S–Ni–S chelate bond angle of 89.12 (2)°. The Ni–S distances [2.2298 (5) and 2.2348 (5) Å] are in agreement with other analogues, such as [Ni(S<sub>2</sub>(POEt)<sub>2</sub>)<sub>2</sub>] [2.230 (4) and 2.236 (4) Å; McConnell & Kastalsky, 1967] and [Ni(S<sub>2</sub>P(OMe)<sub>2</sub>)<sub>2</sub>] [2.225 (5) and 2.219 (2) Å; Kastalsky & McConnell, 1969]. The other bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable with other square-planar nickel(II) complexes. The NiS<sub>4</sub>P<sub>2</sub> fragment is essentially planar with a maximum deviation of 0.153 (1) Å for atom S2 from the mean plane. The interatomic distance between Ni1 and P1 of 2.7914 (6) Å is shorter than that of the octahedral [Niphen(S<sub>2</sub>PO<sub>2</sub>C<sub>6</sub>H<sub>14</sub>)<sub>2</sub>] [3.022 (1) and 2.969 (2) Å; Hao *et al.*, 2001]. The ester fragments, O1/C1–C7 and O2/C8–C14, are essentially planar, with maximum deviations of 0.157 (1) Å for O1 and 0.030 (1) Å for O2, respectively. The two fragments make an angle with each other of 61.10 (11)°.



## Experimental

Na<sub>2</sub>[S<sub>2</sub>PO<sub>2</sub>C<sub>6</sub>H<sub>14</sub>]<sub>2</sub> was first prepared according to the method described in the literature (Kabachnik & Mastyukova, 1953). To a warm 30 ml deionized water solution of Na<sub>2</sub>[S<sub>2</sub>PO<sub>2</sub>C<sub>6</sub>H<sub>14</sub>]<sub>2</sub> (6.0 g, 12.7 mmol) was added 20 ml aqueous solution of NiSO<sub>4</sub> (1.6 g, 10.3 mmol). The mixture was stirred for 20 min and refluxed for 30 min. The solution was filtered and the purple precipitate was washed and dried under vacuum. Crystals suitable for X-ray investigation were obtained by recrystallization from acetone.

Received 24 February 2004

Accepted 19 March 2004

Online 31 March 2004

Crystal data

[Ni(C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>PS<sub>2</sub>)<sub>2</sub>]  
*M<sub>r</sub>* = 677.39  
 Triclinic, *P* $\bar{1}$   
*a* = 7.1884 (11) Å  
*b* = 9.3158 (14) Å  
*c* = 12.1699 (18) Å  
 $\alpha$  = 78.605 (2)°  
 $\beta$  = 81.335 (2)°  
 $\gamma$  = 78.557 (2)°  
*V* = 777.7 (2) Å<sup>3</sup>

*Z* = 1  
*D<sub>x</sub>* = 1.446 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 4396 reflections  
 $\theta$  = 1.7–26.0°  
 $\mu$  = 1.03 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, purple  
 0.48 × 0.26 × 0.23 mm

Data collection

Bruker SMART APEX diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.638, *T<sub>max</sub>* = 0.798  
 8064 measured reflections

3031 independent reflections  
 2789 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.016  
 $\theta_{max}$  = 26.0°  
*h* = -8 → 8  
*k* = -11 → 11  
*l* = -14 → 14

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.030  
*wR*(*F*<sup>2</sup>) = 0.083  
*S* = 1.04  
 3031 reflections  
 180 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.1517P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.35 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.19 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1–S2	2.2298 (5)	S2–P1	1.9743 (7)
Ni1–S1	2.2348 (5)	P1–O1	1.5830 (13)
S1–P1	1.9782 (7)	P1–O2	1.5877 (14)
S2–Ni1–S1	89.12 (2)	O1–P1–O2	99.39 (7)
P1–S1–Ni1	82.75 (2)	S2–P1–S1	104.85 (3)
P1–S2–Ni1	82.97 (2)		

After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atom, with C–H = 0.93–0.96 Å and *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C) or 1.2*U*<sub>eq</sub>(C), respectively.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

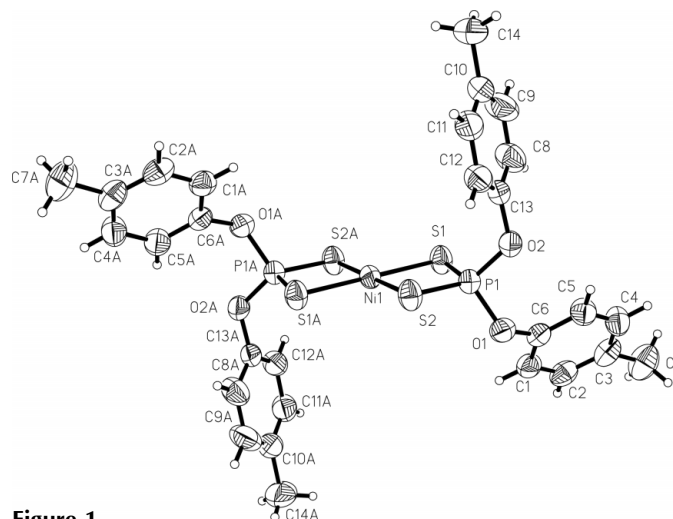


Figure 1

The molecular structure of the title compound, with 50% probability displacement ellipsoids. [Symmetry code: (A) 2 - *x*, 1 - *y*, -*z*.]

This project was supported by the Natural Science Foundation of China (No. 20275020), the Natural Science Foundation of Shandong Province (No. Z2002B02) and the outstanding Adult–Young Scientific Research Encouraging Foundation of Shandong Province (No. 03BS081). The authors also thank the Malaysian Government and Universiti Kebangsaan Malaysia for research grant IRPA No. 09-02-02-0163.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
 Hao, Q., Fun, H.-K., Chantrapromma, S., Razak, I. A., Jian, F., Yang, X., Lu, L. & Wang, X. (2001). *Acta Cryst. C* **57**, 717–718.  
 Jones, J. R. & Symes, T. J. (1971). *J. Chem. Soc. C*, pp. 1124–1130.  
 Kabachnik, M. I. & Mastryukova, T. A. (1953). *Izvest. Akad. Nauk SSSR Otd. Khim. Nauk*, pp. 121–24. (In Russian).  
 Kastalsky, V. & McConnell, J. F. (1969). *Acta Cryst. B* **25**, 909–915.  
 Livingstone, S. E. & Mihkelson, A. E. (1970). *Inorg. Chem.* **9**, 2545–2551.  
 McConnell, J. F. & Kastalsky, V. (1967). *Acta Cryst.* **22**, 853–859.  
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Shiomi, M., Tokashiki, M., Tomizawa, H. & Kurubayashi, T. (1989). *Lubr. Sci.* **1**, 134–137.  
 Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.